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IS 14426 (1997): Chlorinated Paraffins (CP) [PCD 12: Plastics]

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क्लोरीनित पैराफिन (सी पी) – विशिष्टि

Indian Standard

CHLORINATED PARAFFINS (CP) — SPECIFICATION

ICS 71.080.20

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

January 1997

Price Group 3

Plastics Sectional Committee, PCD 12

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastics Sectional Committee, had been approved by the Petroleum, Coal and Related Products Division Council.

Chlorinated paraffins (CP) are chloro-derivatives of normally liquids paraffinic starting materials and having the general formulae $C_xH_{(2x-y+2)}Cl_y$. The properties of a particular commercial chlorinated paraffin depends mainly on three factors, namely, the nature of the paraffinic raw material; the chlorine content and the conditions of manufacture particularly the temperature of chlorination. From any paraffinic raw material, therefore, a series of chlorinated products of progressively changing properties can be obtained by appropriate control of the latter two conditions. In appearance they may be liquid, semifluid or low melting solids. For industrial use ordinarily grades, 40 percent to 70 percent chlorine content of CP are more popular.

The principal raw materials employed in the manufacture of chlorinated paraffin are normal paraffins or paraffin waxes. Chlorinated paraffins are produced by passing gasseous chlorine into the paraffin hydrocarbons at a temperature at which the viscosity of the paraffin is sufficiently low to permit free evolution of hydrochloric acid and solution of the chlorine and decomposition of the chlorinated paraffins is not extensive. Chlorination is generally carried out in the presence of light and in absence of catalyst. Besides impurities, branched chain hydrocarbons and cyclic hydrocarbons present in the raw materials from chlorinated compounds which easily loose hydrochloric acid, cause discoloration and produce products of lesser thermal stability. To improve thermal stability of these chlorinated paraffins, additives such as epoxidised oils, glycols, pentaeryothritol are blended.

The chlorinated paraffins impart properties like non-flammability, electrical properties, heat and light stability, economy and as such they are used as partial replacement of primary plasticizers like di-octyl phthalates (DOP)/di-isooctyl phthalates (DIOP). In fact, the chlorinated paraffins are increasingly used as a secondary plasticizer in PVC-industry, although the compatibility of CP is limited in PVC and depends upon primary plasticizer and its level in the compound, chlorine content of CP, etc. To ensure its non-migration from PVC products within its compatibility limit the product should comply with the specifications of CP of the particular grade.

CP are also used as plasticizer extenders in the processing of vinyl polymers and copolymers, chlorinated rubbers, neoprene, coumarone-indence resins, etc. The incorporation of CP in natural rubber, polyethylene, etc, confers on these flame-retardant characteristics. Because of their viscous nature, compatibility with oils and gradual liberation of hydrochloric acid at high temperature, CP are particularly effective as extreme-pressure lubricant additives and as a boundary lubricant. Various CPs have been used to improve the properties of industrial lubricants such as cutting, drawing and rolling oils. In the paint and various industry, CPs are employed in the compounding of fire-resistant and marine paints as well as modified varnishes.

CPs having high chlorine content (70 percent) are used in the manufacture of dielectric fluids and insulating covering materials for electrical conductors. They are also employed for fire, mould, waterproofing cotton, canvas, ropes and other textile materials. CPs are also used as antirot and anti insect impregnants for wood. CPs in admixture with lead fluoride, polyvinyl chloride and antimony oxide are not only fire-resistant and water repellant, but afford protection against radiation from radioactive sources. Condensation products of CPs with benzene, toluene or the xylenes are excellent lubricants. Presently, there are a number of units manufacturing chlorinated paraffins.

There is a possibility that chlorinated paraffins may be added as plasticizer in the processing of plastic materials which are used for packing and storing of food articles. The committee felt that to cover such usages of chlorinated paraffins, LD 50 values and other requirements should be included in the standard. Since sufficient informations and data are not available for the various requirements to be included, the committee decided to include the same in due course of time.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
CHLORINATED PARAFFINS (CP) — SPECIFICATION

1 SCOPE

This standard prescribes the requirements, methods of sampling and tests for various grades of chlorinated paraffins (CP).

2 NORMATIVE REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1448	Methods of test for petroleum and its products
[P:10] : 1970	Cloud point and pour point (*first revision*)
[P:25] : 1976	Determination of kinematic and dynamic viscosity (*first revision*)
2828 : 1964	Glossary of terms used in plastics industry
9591 : 1996	Methods of sampling and test for plasticizers

3 TERMINOLOGY

For the purpose of this standard definitions given in IS 2828 : 1964 shall apply.

4 GRADES

The material shall be of the following grades:

Grade I — having 40-42 percent chlorine content

Grade II — having 43-46 percent chlorine content

Grade III — having 50-52 percent chlorine content

Grade IV — having 57-58 percent chlorine content

Grade V — having chlorine content of 65 percent and above

5 REQUIREMENTS

5.1 Description

5.1.1 The materials shall be clear liquid having very light or pale yellow colour and shall be free from suspended matter.

5.1.2 The material shall be insoluble in water and soluble in commonly used plasticizers for PVC.

5.1.3 The material shall also be odourless or with slight odour which is not unpleasant.

NOTE — The chlorinated paraffins having above 65 percent chlorine content is solid at ambient temperature and accordingly requirements given in **5.1.1** to **5.1.3** shall be checked on melting the material.

5.2 The materials shall also comply with the requirements given in Table 1.

6 PACKING AND MARKING

6.1 Packing

The containers shall be securely packed in well-closed containers as agreed to between the purchaser and the supplier.

NOTE — Deterioration of colour of the CPs are caused fast by the presence of traces of iron.

6.2 Marking

The containers shall be securely closed and legibly and indelibly marked with the following information:

a) Name and Grade of the material;
b) Indication of the source of manufacture; recognized trade-mark, if any;
c) Tare, net and gross mass;
d) Batch No. or Code No.; and
e) Date of manufacture.

6.3 BIS Certification Marking

The containers may also be marked with the Standard Mark.

The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act,* 1986 and the Rules and Regulations made thereunder. The

Table 1 Requirements for Chlorinated Paraffins

(*Clause* 5.2)

Sl No.	Characteristics	Requirements					Method of Test, Ref to
		Grade I	Grade II	Grade III	Grade IV	Grade V	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Chlorine content, percent (m/m)	40 to 42	43 to 46	50 to 52	57 to 58	65, *Min*	Annex A
ii)	Colour, Hazen, *Max*	125	125	125	150	150	6 of IS 9591 : 1996
iii)	Relative density at 27°C/27°C	1.160 to 1.180	1.210 to 1.230	1.260 to 1.280	1.360 to 1.380	1.450 to 1.480	7 of IS 9591 : 1996
iv)	Absolute viscosity at 27°C, poise, *Min*	1 to 2	3 to 5	12 to 20	40 to 80	To be agreed between the purchaser and the supplier	IS 1448 [P : 25]
v)	Free mineral acidity, mg. KOH/gm, *Max*	0.1	0.1	0.1	0.15	0.25	Annex B
vi)	Pour point, °C, *Min*	– 20	0	10	18	55	IS 1448 [P : 10]
vii)	Volatile loss (after 3 h at 130°C), percent by mass, *Max*	1.5	1.0	0.6	0.5	0.5	Annex C
viii)	Thermal stability index (as percent HCl after 4 h at 175°C), *Max*	0.2	0.2	0.15	0.15	0.2	Annex D

details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

7 SAMPLING AND CRITERIA FOR CONFORMITY

7.1 The method drawing representative samples of the material shall be as prescribed in 4 of IS 9591:1996 using appropriate instruments.

7.2 Number of Tests

7.2.1 Test for the determination of chlorine content shall be conducted on each of the individual samples constituting the set of test samples.

7.2.2 Tests for remaining characteristics shall be conducted on the composite sample.

7.3 Criteria for Conformity

7.3.1 *For Individual Samples*

The mean and the range for the test results for determination of chlorine content shall be calculated as follows:

Mean (X) = The sum of the test results divided by the number of the results.

Range (R) = The difference between the maximum and minimum values of the test results.

7.3.1.1 If the value of the expression ($X - 0.6\,R$) or ($X + 0.6\,R$) as calculated from the test results, lies between the values given in Table 1 for chlorine content, of the sample, the lot shall be declared to have satisfied the requirement of chlorine content.

7.3.2 *For Composite Sample*

The test results on the composite sample shall meet the corresponding requirements.

7.3.3 A lot shall be declared as conforming to the specification, if it satisfies the requirements for each of the characteristics as prescribed under 5.

7.3.4 If the requirements for any of the characteristics are not met, the lot shall be declared to have not satisfied the requirements of this specification.

8 TEST METHODS

Tests for the requirements listed under 5 shall be carried out according to the methods prescribed in IS 9591:1980 and the Annexes of this standard. Reference to the relevant clauses and Annexes are given in col 8 of Table 1.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF CHLORINE CONTENT

A-1 SUMMARY OF METHOD

The sample, dissolved in a low boiling hydrocarbon mixture, is boiled under reflux with metallic sodium and *n*-butyl alcohol. Under these conditions the chlorine is converted to sodium chloride which is then extracted with water. The chloride in the extract is determined volumetrically by titration with silver in the presence of thiocyanate.

A-2 APPARATUS

A-2.1 Reaction Vessel

Consisting of a 300-ml round-bottom flask with standard-taper joint and surmounted with a water-cooled condenser.

NOTE — The condenser tube shall be wide enough to allow rapid reflux without hold-up.

A-3 REAGENTS

A-3.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the relevant Indian Standard specifications wherever available.

A-3.2 Unless otherwise indicated, references to water shall be understood to mean distilled water.

A-3.3 Benzyl Alcohol

The benzyl alcohol used must not show a visible precipitate when tested with alcoholic silver nitrate ($AgNO_3$) solution.

A-3.4 *n*-Butyl Alcohol

A-3.5 Ferric Alum Indicator Solution

Dissolve 350 g of ferric alum [$Fe_2(SO_4)_3.(NH_4)_2.SO_4.24H_2O$] in 1 litre of water and add 200 ml of aerated HNO_3 (1:2).

A-3.6 Isoamyl Alcohol

The isoamyl alcohol used must not show a visible precipitate when tested with alcoholic $AgNO_3$ solution.

A-3.7 Naphtha

Paraffinic, distilling between 99 and 121°C, or petroleum spirit - 100/120.

A-3.8 Nitric Acid (1:7)

Mix 1 volume of concentrated nitric acid (HNO_3, relative density 1.42) with 7 volume of water. Aerate to remove oxides of nitrogen.

A-3.9 Phenolphthalein Indicator Solution (10 g per litre)

Dissolve 1 g of phenolphthalein in iso-propyl alcohol (60 percent) and dilute to 100 ml with the alcohol.

A-3.10 Silver Nitrate, Standard Solution (0.100 0 N)

Dissolve 16.989 0 g of dry (1 h at 110°C) silver nitrate ($AgNO_3$) in water and dilute to 1 litre in a volumetric flask with water.

A-3.11 Sodium, Metal

A-3.12 Sodium Hydroxide Solution (80 g per litre)

Dissolve 80 g of sodium hydroxide (NaOH) in water and dilute to 1 litre.

A-3.13 Thiocyanate, Standard Solution (0.05 N)

Dissolve 3.80 g of ammonium thiocyanate (NH_4CNS) or 4.85 g of potassium thiocyanate (KCNS) crystals in water and dilute to 1 litre. To standardize this solution, measure 40 ml into a flask containing 5 ml of HNO_3 (1:7) and 50 to 60 ml of water. Swirling the flask constantly, measure 22 to 24 ml of 0.100 0 N $AgNO_3$ solution from a burette; then stopper and shake for 20 to 30 seconds. Add 2 to 3 ml of ferric alum indicator solution and titrate with the thiocyanate solution to a red end point. Calculate the normality of the thiocyanate solution as follows:

$$\text{Normality of thiocyanate solution} = \frac{AN}{B}$$

where

A = millilitres of $AgNO_3$ solution;
N = normality of the $AgNO_3$ solution; and
B = total millilitres of the thiocyanate solution.

A-4 PROCEDURE

A-4.1 Shake the sample, if liquid, to ensure homogeneity. Weigh a portion, estimated to contain 0.05 to 0.1 g of chloride, into the 300-ml flask. Add 25 ml of naphtha or petroleum spirit and swirl until the sample is dissolved.

A-4.2 Reaction and Extraction

A-4.2.1 To the solution of the sample, add 5 ml of *n*–butyl alcohol and 2.8 ± 0.2 g of freshly cut sodium. Connect the flask to the condenser and boil the liquid vigorously under reflux for 1 h. Introduce an additional 20 ml of *n*-butyl alcohol through the top of the condenser and continue refluxing until no metallic sodium remains.

A-4.2.2 Add 25 ml of water to the flask through the condenser, cool to room temperature, and transfer the mixture to a 500-ml separating funnel. Wash the flask with 50 ml of HNO_3 (1:7) and then with 50 ml of naphtha or petroleum spirit. Add these washings to the separating funnel. Adjust the acidity of the aqueous layer until just acidic to phenolphthalein, using HNO_3 (1:7) or NaOH (80 g per litre) reagents for this purpose; then add 12 to 13 ml of HNO_3 (1:7).

A-4.2.3 Shake the funnel and contents, and after settling, draw the aqueous layer into a clean 500-ml glass stoppered Erlenmeyer flask. Wash the original 300-ml reaction flask with 25 ml of water, transfer to the separating funnel, wash the naphtha or petroleum spirit layer which remained in the separatory funnel after separating the aqueous layer; transfer this washing to the 500-ml flask. Repeat with a second 25-ml portion of water.

A-4.3 Chloride Determination

A-4.3.1 Boil the combined acidic aqueous extracts and washings until free of hydrogen sulphide (H_2S) as detected by dipping a piece of lead acetate test paper into the solution (no black discoloration). If the solution is clear, proceed with the directions as given in **A-4.3.2** and **A-4.3.3**; if not filter the solution through a qualitative filter paper into another 500-ml glass-stoppered Erlenmeyer flask. Rinse the original flask with two 25-ml portions of water, using each portion to wash the filter paper.

A-4.3.2 After the solution has reached room temperature, add 10 ml of *iso*-amyl alcohol or benzyl alcohol (*see* Note), 3 ml of ferric alum indicator and, from a burette, 0.4 to 0.6 ml of 0.05 N thiocyanate solution. Swirling constantly, titrate with 0.1 N $AgNO_3$ solution until the red colour is discharged; then add 2 to 5 ml in excess.

NOTE — Because of the extreme toxicity of nitrobenzene, its use has been discontinued, and the use of either isoamyl alcohol or benzyl alcohol is recommended. Since both of these alcohols are said by some authorities to have very moderate toxic properties, care should be exercised with their use, but they are relatively many times less toxic than nitrobenzene.

A-4.3.3 Stopper the flask tightly and shake vigorously for 15 s to coagulate and remove the precipitate from the aqueous phase (CAUTION, Note). Without refilling the burette, titrate slowly with 0.05 N thiocyanate solution until the end point is approached, as indicated by a more slowly fading red colour. Stopper the flask, shake vigorously for 20 to 30 s, and continue the titration until one drop produces a distinct reddish coloration that does not fade upon swirling or vigorous shaking.

NOTE — CAUTION — Care should be exercised in opening the flask after shaking. Pressure may develop, causing small amounts of the acid solution to be sprayed from the mouth of the flask as the stopper is removed.

A-4.3.4 *Blank Determinations*

Simultaneously with each set of sample determinations, make duplicate blank determinations, following exactly the procedure used for the sample determinations (except for ommission of the sample). Always use identical reagents for the blank determination and for the sample determination. If the titration exceeds 0.05 ml, discard the responsible reagent.

A-5 CALCULATION

Calculate the total chlorine content of the sample as follows:

Chlorine content, percent by mass

$$= \frac{(AB - CD)\times 35.46}{10\,M}$$

where

A = millilitres of $AgNO_3$ solution,
B = normality of the $AgNO_3$ solution,
C = millilitres of thiocyanate solution used,
D = normality of the thiocyanate solution, and
M = grams of original sample used.

A-6 REPORT

Report the result to the nearest 0.01 percent as the chlorine content.

A-7 PRECISION

The following criteria should be used for judging the acceptability of results (95 percent confidence):

a) Repeatability — Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

Chlorine Content, percent	*Repeatability*
0.1 to 1	0.03
Over 1	3 percent of amount present

b) Reproducibility — The result submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amounts:

Chlorine Content, percent	*Repeatability*
0.1 to 1	0.04
Over 1	6 percent of amount present

ANNEX B

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF FREE MINERAL ACIDITY

B-1 OUTLINE OF THE METHOD

Free mineral acidity is extracted in aqueous phase which is titrated with standard solution of sodium hydroxide using bromophenol blue indicator.

B-2 REAGENTS

B-2.1 Toluene

B-2.2 Standard sodium hydroxide solution of 0.1 N.

B-2.3 Bromophenol blue indicator solution.

B-2.4 Sodium chloride solution (20 g in 200 ml of water).

B-3 PROCEDURE

Take 100 g of CP which is dissolved in 100 ml toluene. This solution is extracted with 200 ml water containing 20 g of sodium chloride. Titrate 100 ml of water extracted with N/10 sodium hydroxide solution with bromophenol blue indicator.

B-4 CALCULATION

Mineral acidity

$$\text{(mg KOH/g of CP)} = \frac{56.1 \times 0.1 \times 2\,V}{100}$$

where

V = volume of sodium hydroxide solution required.

ANNEX C

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF VOLATILITY

C-1 OUTLINE OF THE METHOD

Volatility is measured as percentage loss of weight for 10 g of plasticizer used when heated in oven at 130 ± 2°C for 3 hours.

C-2 APPARATUS

C-2.1 Oven, convection type, capable of being controlled at 130 ± 2°C.

C-2.2 Petridish, glass, 75 mm diameter.

C-2.3 Desiccator

C-2.4 Asbestos disc, 2-3 mm thick and 200 mm in diameter.

C-3 PROCEDURE

C-3.1 From the same lot draw two samples A and B and then weigh accurately two samples from A and two samples from B each about 10 g in the petridish. Place all the four Petridishes on the asbestos disc and keep them in the oven controlled at 130 ± 2°C for 3 h. Remove the petridishes and cool in a desiccator.

C-3.2 Weigh to the nearest 0.1 mg.

C-4 EXPRESSION OF RESULTS

Volatile loss, as percentage by mass, is given by the formula:

$$\text{Volatile loss, percent} = \frac{m_1 - m_2}{m_1} \times 100$$

where

m_1 = mass in g of the test portion, and

m_2 = mass in g of the residue.

Take average value of the two readings. If the two values differ remarkably test shall be repeated.

ANNEX D

[*Table* 1, *Sl No.* 9 (viii)]

DETERMINATION OF THERMAL STABILITY

D-1 OUTLINE OF THE METHOD

Hydrochloric acid formed by decomposition of the product by application of heat is measured.

D-2 APPARATUS

D-2.1 Two necked flask of 100 ml capacity.

D-2.2 Electrically heated oil to maintain temperature 175 ± 5°C.

D-2.3 Nitrogen

D-2.4 Flow meter for nitrogen with a capacity of 10 litres per hour.

D-2.5 Absorption bottle of 250 ml capacity.

D-3 PROCEDURE

Weigh 30 g of the sample in a two necked flask. Place the flask in oil bath. Connect nitrogen tube through flow meter to the flask. Similarly connect outlet nozzle by tube to absorption flask containing 100 ml water. Heat the product to 175 ± 2°C and pass nitrogen at the rate of 10 litres/hour for 4 hours. Hydrogen chloride generated is absorbed in water which is titrated against N/10 sodium hydroxide solution.

D-4 CALCULATION

Amount of hydrogen chloride evolved is calculated according to following equation and expressed as gram per 100 g of the product as thermal stability index:

$$= \frac{\text{CC of N/10 NaOH} \times 36}{\text{Wt of Product}}$$

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